

STUDY OF COMPOSITION AND HOMOGENITY OF $\text{Ni}_{80}\text{Fe}_{20}$ PERMALLOY IN THE FORM OF TARGET AND THIN FILM AS MATERIALS OF MAGNETIC SENSORS

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ABSTRACT

STUDY OF COMPOSITION AND HOMOGENITY OF $\text{Ni}_{80}\text{Fe}_{20}$ PERMALLOY IN THE FORM OF TARGET AND THIN FILM AS MATERIALS OF MAGNETIC SENSORS. Material fabrication of a ferromagnetic permalloy $\text{Ni}_{80}\text{Fe}_{20}$ has successfully been done. The sample was used as a target in magnetic thin film deposition by the sputtering method. The corresponding characterization including spectral analysis with XRF and XRD supports the prediction that the permalloy has been formed. The XRF analysis result shows that the ratio of the target has good agreement with the calculation. The thin film produced by sputtering gave the same characteristic as the target. From the XRD analysis it was shown that $\text{Ni}_{80}\text{Fe}_{20}$ permalloy was formed in the target and thin film, they were shown with the peak at the angle of $2\theta = 44,4^\circ$, at a (111) plane orientation as the hkl plane from $\text{Ni}_{80}\text{Fe}_{20}$. There is another peak at the angle $2\theta = 37^\circ$ that was estimated to be ferrosic oxide Fe_3O_4 . This magnetic oxide was formed when the target had been heated at the temperature 350°C - 400°C in the free air.

Key words : $\text{Ni}_{80}\text{Fe}_{20}$ permalloy, thin film, magnetic sensor materials

ABSTRAK

STUDI KOMPOSISI AND HOMOGENITAS PERMALLOY $\text{Ni}_{80}\text{Fe}_{20}$ DALAM BENTUK TARGET DAN LAPISAN TIPIS SEBAGAI BAHAN DASAR SENSOR MAGNET. Telah berhasil dilakukan pembuatan bahan paduan *ferromagnetic permalloy* $\text{Ni}_{80}\text{Fe}_{20}$. Bahan Tersebut digunakan sebagai bahan target dalam proses pendeposisian lapisan tipis magnetik dengan metode *sputtering*. Karakterisasi yang mencakup analisis difraktogram XRF dan XRD membenarkan dugaan telah terbentuk paduan tersebut. Dari hasil analisis XRF bahwa perbandingan target telah sesuai dengan perhitungan. Hasil lapisan tipis juga telah terbukti sesuai dengan target. Sedangkan dari analisis XRD menunjukkan bahwa pada target dan lapisan tipis telah terbentuk *permalloy* $\text{Ni}_{80}\text{Fe}_{20}$, ini nampak adanya puncak pada sudut $2\theta = 44,4^\circ$, dengan orientasi bidang hkl (111) yang merupakan bidang hkl dari $\text{Ni}_{80}\text{Fe}_{20}$. Terlihat pula adanya puncak lain pada sudut $2\theta = 37^\circ$ disinyalir sebagai oksida ferosik (*ferrosic oxide*) Fe_3O_4 . Oksida yang bersifat magnetik ini terbentuk saat target dipanaskan pada suhu 350°C - 400°C di udara bebas.

Kata kunci : *Permalloy* $\text{Ni}_{80}\text{Fe}_{20}$, lapisan tipis, bahan sensor magnet

INTRODUCTION

Thin film technology application has reached all aspects recently. Magnetic thin film has become research object that is interesting for researcher. This is because the results can be used as basic for the development of microelectronic technology. For example, the thin film can be used as the material of magnetic transistor [1] and magnetoresistive sensor [2].

The magnetoresistive sensor constitutes a sensor that uses a change of resistance caused by a change of magnetic field, therefore it includes magnetic sensors. Since resistance is the only property involved measurement can be done using a simple electronic instrument.

The first sensor that uses the effect was found 130 years ago. But that discovery was first used just after 100 years. Later motivated by need of a very sensitive magnetic sensor and the development of thin film fabrication technology, exploitation on magnetoresistive effect.

The thin film used as magnetic sensor must be deposited from magnetic field sensitive materials, and the ferromagnetic materials is a suitable materials required. In this research, the researchers will investigate the fabrication of $\text{Ni}_{80}\text{Fe}_{20}$ alloy materials. The alloy material is chosen because it does not show any sign of magnetostriction, so that it is unnecessary to take into

account the appearance of the change of lattice structure caused by magnetic field.

The permalloy responds magnetic field that derives from a very fast current change [3]. The other reason is the materials show high enough magnetoresistive effect in the form of thin film, which is 2.2% [4]. The research is therefore designed to obtain a sensitive magnetic sensor.

In this research, the sputtering method is chosen to deposit permalloy on a substrate of microscope slide glass, because the used materials have a high enough melting point, it is difficult to use other method. Apart from that the thin film formed by sputtering method is known to have the same composition with the composition of target materials [5]. And finally, it is known that the deposition taking place during sputtering process is easier to control.

The objectives of this research is to compare between the composition and the structure of target material to those of thin film. The result of this research is expected to give a complete comprehension of the thin film fabrication technology as the basic material for magnetoresistive sensor and any physical parameters of the thin film which are related to the magnetoresistive sensor.

METHOD

Materials used as the targets material are Ni and Fe in the form of powder with the grain size smaller than 10 micrometers and purity of 99.5 % (the other contents are antei 0.1% ; chloride 0.002 %; nitrogen 0.002%, ; Pb 0.01 %; Cu 0.02%). Substrate material is microscope slide glass with the width of 10 mm, length of 25 mm and thickness of 1 mm while the plasma is formed by argon gases. The molding device of the target has the diameter of 60 mm and the height of 50 mm, it is made of SS304 material. (it is used to produce the target). A press equipments has the capability until 20 ton (it is used to press the target). The furnace used can operate till 2000°C (for heating target).The sputtering is used for the thin film deposition. The equipment for characterization comprise XRD with Cu cathode source (it is used for structural characterization) and XRF with Cd109 Excitation source (it is used for homogeneity test)

The experiment is done in three steps. (i) the $\text{Ni}_{80}\text{Fe}_{20}$ permalloy target material fabrication , (ii) Thin film deposition with sputtering method, (iii)The characterization of composition and structure with XRF and XRD devices.

Target fabrication is begun with the weighing of material. The material needed is 39.65 gram Ni and 9.43 gram Fe (for the comparison of 80% and 20 %) for the target size with the diameter of 60mm and thickness of 2 mm. Both materials are mixed thoroughly. After that it is brought into the furnace at the temperature of 400°C for 10 minutes. Furthermore, it is poured into the mold

and pressed by the load of 16 ton for 3 minutes, and followed by heat treatment at 400°C for another 10 minutes.

The substrate is made of the microscope slide glass with the of size 1 cm x 2.5 cm and the thickness of 1mm. The substrate is washed by detergent. Then, it is cleaned by aquades and alcohol 95%. Furthermore it is brought in to the ultrasonic cleaner for 5 minutes and finally it is dried.

The process of the $\text{Ni}_{80}\text{Fe}_{20}$ permalloy deposition, is started by putting the clean glass substrate on an anode in the reactor tube. The reactor tube is then vacuumed by a rotary pump to go to the pressure in the order of 10^{-2} Torr. After that, the heater system and the diffusion pump are switched on until the pressure reaches the order of 10^{-5} Torr. Furthermore the argon gas is flowed in to the reactor until the pressure reaches 5.10^{-2} Torr. Finally it switches on the DC voltage source suitable with the working voltage that is 1.4 Kilovolt and current 16mA.

The XRF is used to find the composition of the thin film and the target that is made, while the XRD is used to fine the structure of the target and the thin film.

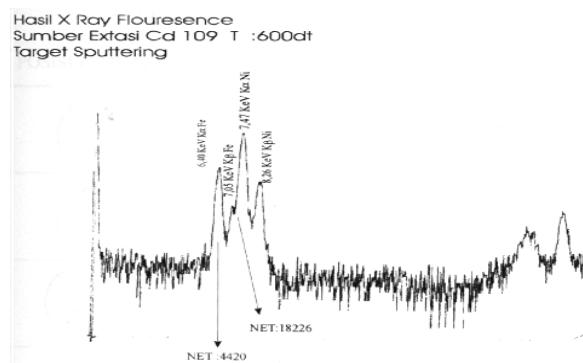


Figure 1. The result of spectrum XRF (the test of target homogeneity)

RESULT AND DISCUSSION






The target obtained has the thickness of 3 mm and the diameter of 60 mm. The experiment result in thickness is different from the calculation, i.e. 2 mm. This might be due to the target pressure which is not strong enough, therefore the material is not compact.

The homogeneity of the target was examined by X- Ray Flourescence (XRF) method. The homogeneity of target was examined at five different points (Table 1). The Table 1 indicates that the target is homogeneous enough because the ratio of Fe: Ni at different positions is similar, that is 1: 4.

The XRD analysis was carried out to find the crystal structure of the target, and the result can be seen in Figure 2. From Figure 2, the XRD result for the target material shows the peak at the angle $2\theta = 44.3^\circ$ and from the calculation it is known as the (111) hkl plane, this is the peak of $\text{Ni}_{80}\text{Fe}_{20}$, therefore it can be

concluded that the material target is composed of the $\text{Ni}_{80}\text{Fe}_{20}$ alloy.

Table 1. The result of the test of target homogeneity

| Counter position | Count | Energy |
|---|---|----------------------------|
|  | NET Fe:4420 NET Ni:18226 Fe:Ni = 1:4.1 | Fe:6.40keV Ni:7.47 keV |
|  | NET Fe:4387 NET Ni:18107 Fe:Ni = 1: 4.1 | Fe:6.40 keV Ni:7.47 keV |
|  | NET Fe : 4483 NET Ni:18188 Fe: Ni = 1 : 4 | Fe:6.40 keV Ni:7.47 keV |
|  | NET Fe:4442 NET Ni:18131 Fe : Ni =1: 4 | Fe:6.40 keV Ni:7.47 keV |
|  | NET Fe:4369 NET Ni:17972 Fe:Ni=1: 4 | Fe:6.40 keV Ni:7.47 keV |

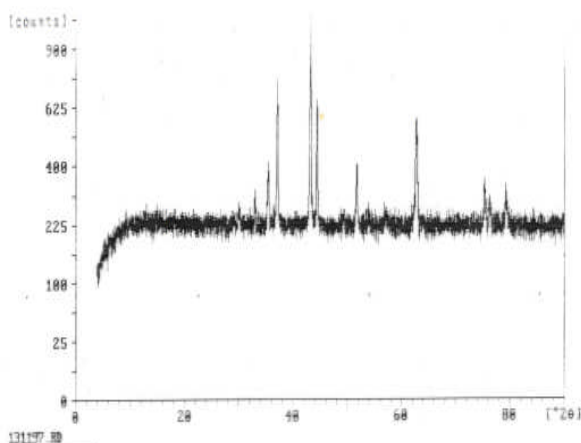


Figure 2. The XRD result for the target material

The thin film resulted by sputtering method with the variation of deposition time is tested by XRF. The XRF test is to find the thin film composition and homogeneity. Figure 3 shows the result of XRF test. From Figure 3, the data as given in Table 2 can be obtained.

The result of the XRF analysis on the deposited glass is compared with that on the non-deposited glass. There are two peaks appear in the deposited alloy, that is the presence of the peaks at the energy of 6.4 keV and 7.47 keV associated with the peak $K\alpha$ of Ni and Fe elements. Therefore, it can be concluded that the film obtained contains Ni and Fe.

From the spectrum it can also be seen peak at the energy of 15.77 keV that constitutes the energy Zr, it constitutes the element of substrate that is used,

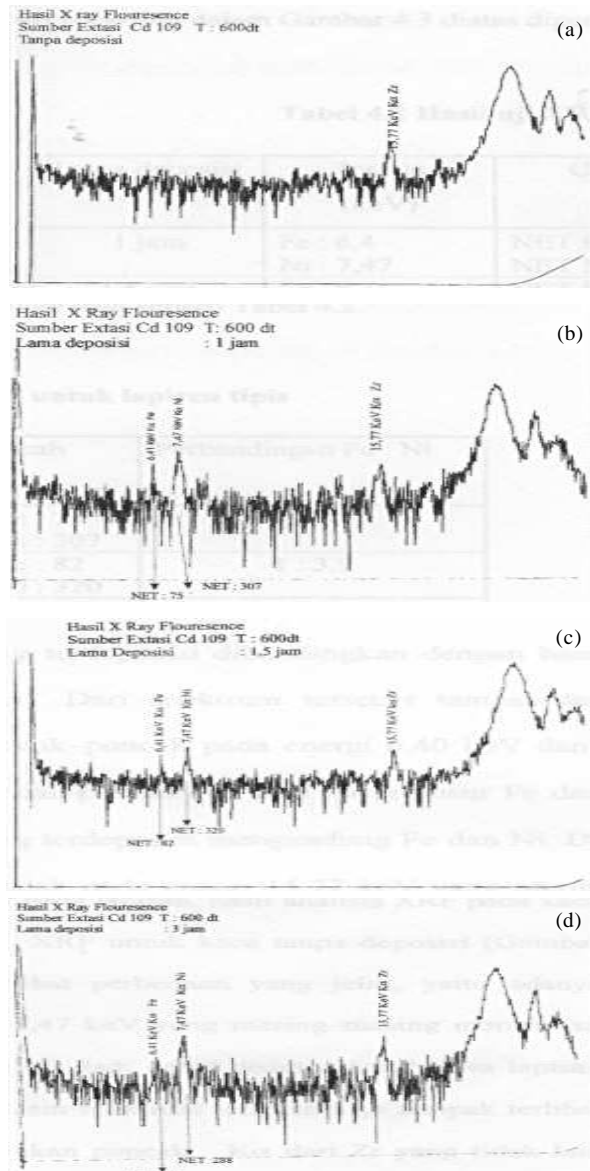


Figure 3. The result of thin film XRF test with time variation: (a) non deposition, (b) 1 hour (c) 1 hour 30 minutes and (d) 3 hours

Table 2. The result of the test of thin film homogeneity

| Time deposition | Energy (K eV) | Count | Comparison Ni : Fe |
|-----------------|---------------------|---------------------------|--------------------|
| 1 hours | Fe : 6.4 Ni:7.47 | NET Fe : 75 NET Ni:307 | 4 : 1 |
| 1,5 hours | Fe : 6.4 Ni:7.47 | NET Fe:82 NET Ni:320 | 3.9 : 1 |
| 3 hours | Fe : 6.4 Ni:7.47 | NET Fe:72 NET Ni:288 | 4 : 1 |

therefore the peak of Zr also appeared at the non-deposited glass.

Finally, from the data given in Table 2 it can be concluded that the thin film composition is similar for the target composition, the Ni : Fe ratio is 4 : 1

Figure 4 shows the result of XRD analysis. There is a peak at the angle $2\theta = 44.4^\circ$, the plane index

hkl resulted from $2d \sin \theta = n\lambda$ and $d = a^2/(h^2+k^2+l^2)$ is found to be $\langle 111 \rangle$ it constitutes the plane index hkl from $\text{Ni}_{80}\text{Fe}_{20}$ [6]. Therefore, it can be concluded that the thin film has been formed from $\text{Ni}_{80}\text{Fe}_{20}$ alloy.

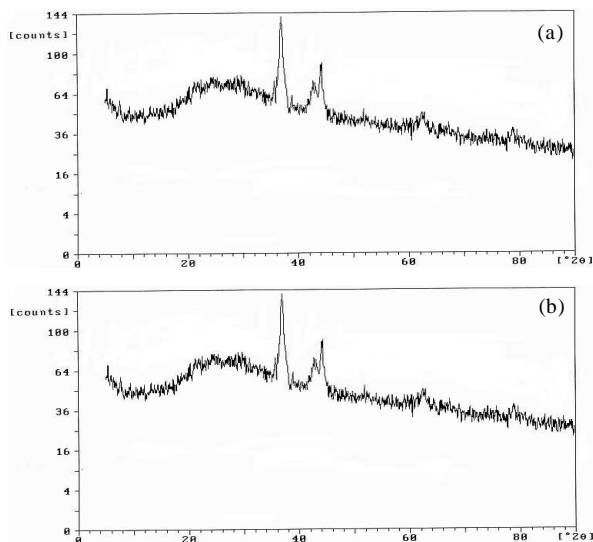


Figure 4. the result of $\text{Ni}_{80}\text{Fe}_{20}$ permalloy thin film XRD analysis with thickness variation (a) 0,64 μm (1 hour) and (b) 1,1 μm (2 hours)

Beside the characteristic peak at the angle $2\theta = 44.4^\circ$, the other peak at the angle $2\theta = 37.77^\circ$, is gone at the thickness of 1,1 micrometers. The peak is estimated from ferrosic oxide Fe_3O_4 . This magnetic Oxide is formed when the target is heated at temperature of $350^\circ\text{--}400^\circ$ [7]. The intensity of the ferrosic oxide spectrum can not be seen at the thickness of 1,1 micrometers, this is caused by the bond of the weak oxide atom. Therefore, the longer deposition time causes the more bond of the oxide atoms are broken [8].

CONCLUSION

The sputtering method is proved to give the $\text{Ni}_{80}\text{Fe}_{20}$ thin film that having the same composition and homogeneity as the target. This is supported by the result of the XRF analysis. The thin film and target studied in this research has been formed from Ni-Fe alloy. This is proved by the XRD test result.

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